

5-Iodo-3-methylsulfinyl-2-phenyl-1-benzofuran

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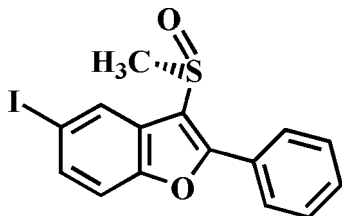
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Key indicators: single-crystal X-ray study; $T = 173$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.017; wR factor = 0.044; data-to-parameter ratio = 15.3.

The title compound, $\text{C}_{15}\text{H}_{11}\text{IO}_2\text{S}$, was prepared by the oxidation of 5-iodo-3-methylsulfanyl-2-phenyl-1-benzofuran using 3-chloroperbenzoic acid. The O atom and the methyl group of the methylsulfinyl substituent lie on opposite sides of the plane of the benzofuran fragment. The phenyl ring is rotated out of the benzofuran plane, with a dihedral angle of $29.84(6)^\circ$. The crystal structure is stabilized by normal intermolecular aromatic π - π stacking interactions with a centroid-to-centroid distance of $3.836(3)$ Å between the furan rings of neighboring molecules, by a weak $\text{C}-\text{H}\cdots\text{O}$ hydrogen bond, and by an $\text{I}\cdots\text{O}$ halogen bond with an $\text{I}\cdots\text{O}$ distance of $3.209(2)$ Å and a nearly linear $\text{C}-\text{I}\cdots\text{O}$ angle of $169.12(6)^\circ$.

Related literature

For the crystal structures of isomers of the title compound, see: Choi *et al.* (2007*a,b*). For a review of halogen bonding, see: Politzer *et al.* (2007). For related literature, see: Allen (2002).



Experimental

Crystal data

$\text{C}_{15}\text{H}_{11}\text{IO}_2\text{S}$	$\gamma = 112.197(1)^\circ$
$M_r = 382.20$	$V = 687.91(6) \text{ \AA}^3$
Triclinic, $P\bar{1}$	$Z = 2$
$a = 8.1495(4) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 8.1823(4) \text{ \AA}$	$\mu = 2.47 \text{ mm}^{-1}$
$c = 11.2007(6) \text{ \AA}$	$T = 173(2) \text{ K}$
$\alpha = 93.584(1)^\circ$	$0.40 \times 0.20 \times 0.10 \text{ mm}$
$\beta = 92.941(1)^\circ$	

Data collection

Bruker SMART CCD area-detector diffractometer	5403 measured reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1999)	2648 independent reflections
$T_{\min} = 0.556$, $T_{\max} = 0.783$	2587 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.014$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.017$	173 parameters
$wR(F^2) = 0.044$	H-atom parameters constrained
$S = 1.09$	$\Delta\rho_{\text{max}} = 0.58 \text{ e \AA}^{-3}$
2648 reflections	$\Delta\rho_{\text{min}} = -0.52 \text{ e \AA}^{-3}$

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C6}-\text{H6}\cdots\text{O1}^i$	0.95	2.59	3.536(2)	173

Symmetry code: (i) $-x + 1, -y, -z + 1$.

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1997); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 (Farrugia, 1997) and DIAMOND (Brandenburg, 1998); software used to prepare material for publication: SHELXL97.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZL2044).

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supplementary materials

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5-Iodo-3-methylsulfinyl-2-phenyl-1-benzofuran

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Comment

As part of our continuing studies on the synthesis and structure of 5-halo-1-benzofuran analogues, we have recently described the crystal structures of 5-chloro-3-methylsulfinyl-2-phenyl-1-benzofuran (Choi *et al.*, 2007*a*) and 5-bromo-3-methylsulfinyl-2-phenyl-1-benzofuran (Choi *et al.*, 2007*b*). Herein we report the molecular and crystal structure of the title compound, 5-iodo-3-methylsulfinyl-2-phenyl-1-benzofuran (Fig. 1).

The benzofuran unit is essentially planar, with a mean deviation of 0.012 Å from the least-squares plane defined by the nine constituent atoms. In the title compound, the dihedral angle between the planes of the benzofuran and the phenyl ring is 29.84 (6)°. The molecular packing (Fig. 2) is stabilized by $\pi\cdots\pi$ stacking interactions between adjacent furan units. The $Cg\cdots Cg^i$ distance is 3.836 (3) Å (Cg is the centroid of the O1/C8/C1/C2/C7 furan ring; symmetry code as in Fig. 2), which is in the usual range when compared to other such interactions (Choi *et al.*, 2007*a, b*). The molecular packing (Fig. 2) is further stabilized by a weak C—H \cdots O hydrogen bond (Table 1) and by a halogen bond between the iodine atom and the oxygen of the S=O unit. This type of strong electrostatic interaction, which is often compared to hydrogen bonding, relies on an unusually strong attraction between the positively charged outermost region of the halogen atom (the " σ -hole") and one of the lone pairs of the acceptor, here at the S=O oxygen atom (Politzer *et al.*, 2007). The observed I \cdots O separation in the structure of the title compound of 3.209 (2) Å and also the nearly linear C—I \cdots O angle of 169.12 (6)° are in the typical range for a such a halogen bond and compare well with values of similar compounds found in the 2007 release of the Cambridge Crystallographic Database V5.28 (Allen, 2002), which revealed 39 compounds with C—I \cdots O=S contacts equal or less than 3.3 Å.

Experimental

3-Chloroperbenzoic acid (77%, 359 mg, 1.60 mmol) was added in small portions to a stirred solution of 5-iodo-3-methylsulfonyl-2-phenyl-1-benzofuran (549 mg, 1.50 mmol) in dichloromethane (30 ml) at 273 K. After being stirred at room temperature for 2 h, the mixture was washed with saturated sodium bicarbonate solution and the organic layer was separated, dried over magnesium sulfate, filtered and concentrated in vacuum. The residue was purified by column chromatography (ethyl acetate) to afford the title compound as a colorless solid [yield 84%, m.p. 464–465 K; R_f = 0.70 (hexane-ethyl acetate, 1:2 v/v)]. Single crystals suitable for X-ray diffraction were prepared by slow evaporation of a dilute solution of the title compound in tetrahydrofuran at room temperature.

Refinement

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.95 Å for aromatic H atoms and 0.98 Å for methyl H atoms, respectively, and with $U_{iso}(H) = 1.2U_{eq}(C)$ for aromatic H atoms and $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl H atoms. The highest peak in the difference map is 1.29 Å from S and the largest hole is 0.67 Å from I.

Figures

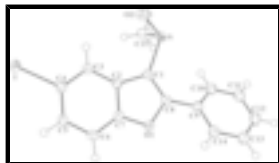


Fig. 1. The molecular structure of the title compound, showing displacement ellipsoids drawn at the 50% probability level.

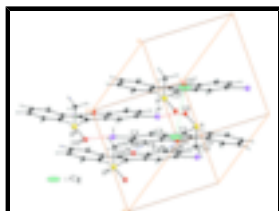


Fig. 2. The π - π , C—H \cdots O hydrogen bond, and I \cdots O halogen bond (dotted lines) in the title compound. Cg denotes the ring centroid. [Symmetry code: (i) $1 - x, 1 - y, 1 - z$; (ii) $1 - x, -y, -z$; (iii) $1 - x, -y, 1 - z$.]

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Crystal data

$C_{15}H_{11}IO_2S$	$Z = 2$
$M_r = 382.20$	$F_{000} = 372$
Triclinic, $P\bar{1}$	$D_x = 1.845 \text{ Mg m}^{-3}$
Hall symbol: -p 1	Mo $K\alpha$ radiation
$a = 8.1495(4) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$b = 8.1823(4) \text{ \AA}$	Cell parameters from 5532 reflections
$c = 11.2007(6) \text{ \AA}$	$\theta = 2.7\text{--}28.3^\circ$
$\alpha = 93.584(1)^\circ$	$\mu = 2.47 \text{ mm}^{-1}$
$\beta = 92.941(1)^\circ$	$T = 173(2) \text{ K}$
$\gamma = 112.197(1)^\circ$	Block, colourless
$V = 687.91(6) \text{ \AA}^3$	$0.40 \times 0.20 \times 0.10 \text{ mm}$

Data collection

Bruker SMART CCD area-detector diffractometer	2648 independent reflections
Radiation source: fine-focus sealed tube	2587 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.014$
Detector resolution: $10.00 \text{ pixels mm}^{-1}$	$\theta_{\text{max}} = 26.0^\circ$
$T = 173(2) \text{ K}$	$\theta_{\text{min}} = 1.8^\circ$
φ and ω scans	$h = -9 \rightarrow 10$
Absorption correction: multi-scan (SADABS; Sheldrick, 1999)	$k = -10 \rightarrow 10$
$T_{\text{min}} = 0.556, T_{\text{max}} = 0.783$	$l = -13 \rightarrow 13$
5403 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.017$	H-atom parameters constrained
$wR(F^2) = 0.044$	$w = 1/[\sigma^2(F_o^2) + (0.0213P)^2 + 0.4423P]$
$S = 1.09$	where $P = (F_o^2 + 2F_c^2)/3$
2648 reflections	$(\Delta/\sigma)_{\max} < 0.001$
173 parameters	$\Delta\rho_{\max} = 0.58 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\min} = -0.52 \text{ e } \text{\AA}^{-3}$
	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
I	0.380751 (18)	0.189438 (16)	-0.025139 (11)	0.02710 (6)
S	0.80496 (6)	0.78490 (6)	0.39542 (4)	0.02225 (10)
O1	0.64855 (18)	0.30463 (17)	0.51101 (12)	0.0222 (3)
O2	0.67054 (19)	0.80987 (19)	0.31109 (14)	0.0296 (3)
C1	0.7264 (2)	0.5567 (2)	0.41861 (17)	0.0205 (4)
C2	0.6197 (2)	0.4117 (2)	0.33198 (17)	0.0193 (4)
C3	0.5622 (3)	0.3940 (3)	0.21014 (17)	0.0215 (4)
H3	0.5904	0.4940	0.1648	0.026*
C4	0.4628 (3)	0.2248 (3)	0.15866 (17)	0.0221 (4)
C5	0.4178 (3)	0.0753 (3)	0.22367 (18)	0.0237 (4)
H5	0.3480	-0.0384	0.1848	0.028*
C6	0.4743 (3)	0.0916 (3)	0.34438 (18)	0.0239 (4)
H6	0.4456	-0.0082	0.3901	0.029*
C7	0.5747 (3)	0.2610 (3)	0.39414 (17)	0.0205 (4)
C8	0.7399 (2)	0.4864 (2)	0.52391 (18)	0.0205 (4)
C9	0.8318 (3)	0.5574 (3)	0.64242 (18)	0.0212 (4)
C10	0.9897 (3)	0.7066 (3)	0.65581 (19)	0.0253 (4)
H10	1.0377	0.7645	0.5873	0.030*

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C11	1.0773 (3)	0.7714 (3)	0.7686 (2)	0.0293 (4)
H11	1.1846	0.8737	0.7773	0.035*
C12	1.0082 (3)	0.6868 (3)	0.86842 (19)	0.0309 (5)
H12	1.0683	0.7311	0.9456	0.037*
C13	0.8517 (3)	0.5375 (3)	0.85625 (19)	0.0291 (4)
H13	0.8051	0.4800	0.9252	0.035*
C14	0.7626 (3)	0.4716 (3)	0.74390 (18)	0.0244 (4)
H14	0.6554	0.3692	0.7358	0.029*
C15	0.9862 (3)	0.7957 (3)	0.3080 (2)	0.0364 (5)
H15A	0.9416	0.7108	0.2369	0.055*
H15B	1.0735	0.7666	0.3564	0.055*
H15C	1.0428	0.9157	0.2830	0.055*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I	0.03718 (9)	0.02394 (8)	0.01870 (8)	0.01122 (6)	-0.00362 (5)	-0.00127 (5)
S	0.0234 (2)	0.0155 (2)	0.0264 (2)	0.00579 (18)	0.00115 (18)	0.00217 (18)
O1	0.0271 (7)	0.0176 (7)	0.0184 (7)	0.0050 (5)	-0.0008 (5)	0.0021 (5)
O2	0.0276 (7)	0.0255 (7)	0.0369 (8)	0.0106 (6)	0.0000 (6)	0.0103 (6)
C1	0.0217 (9)	0.0162 (9)	0.0224 (9)	0.0061 (7)	0.0012 (7)	0.0017 (7)
C2	0.0195 (9)	0.0164 (9)	0.0212 (9)	0.0061 (7)	0.0012 (7)	0.0007 (7)
C3	0.0262 (10)	0.0182 (9)	0.0206 (9)	0.0090 (8)	0.0014 (7)	0.0035 (7)
C4	0.0255 (9)	0.0240 (10)	0.0170 (9)	0.0103 (8)	-0.0004 (7)	0.0004 (7)
C5	0.0258 (10)	0.0174 (9)	0.0238 (10)	0.0045 (7)	0.0008 (8)	-0.0016 (8)
C6	0.0289 (10)	0.0171 (9)	0.0237 (10)	0.0059 (8)	0.0028 (8)	0.0052 (8)
C7	0.0227 (9)	0.0214 (9)	0.0173 (9)	0.0083 (7)	0.0016 (7)	0.0020 (7)
C8	0.0202 (9)	0.0169 (9)	0.0232 (10)	0.0059 (7)	0.0021 (7)	0.0008 (7)
C9	0.0235 (9)	0.0218 (9)	0.0202 (9)	0.0111 (8)	0.0002 (7)	0.0013 (7)
C10	0.0244 (10)	0.0256 (10)	0.0245 (10)	0.0081 (8)	0.0003 (8)	0.0022 (8)
C11	0.0248 (10)	0.0276 (11)	0.0306 (11)	0.0061 (8)	-0.0043 (8)	-0.0029 (9)
C12	0.0325 (11)	0.0349 (12)	0.0234 (10)	0.0126 (9)	-0.0063 (8)	-0.0038 (9)
C13	0.0334 (11)	0.0335 (11)	0.0209 (10)	0.0133 (9)	0.0017 (8)	0.0031 (8)
C14	0.0254 (10)	0.0231 (10)	0.0240 (10)	0.0088 (8)	0.0009 (8)	0.0013 (8)
C15	0.0295 (11)	0.0360 (12)	0.0496 (14)	0.0155 (10)	0.0155 (10)	0.0161 (11)

Geometric parameters (\AA , $^\circ$)

I—C4	2.099 (2)	C6—H6	0.9500
I—O2 ⁱ	3.209 (2)	C8—C9	1.461 (3)
S—O2	1.490 (2)	C9—C10	1.392 (3)
S—C1	1.771 (2)	C9—C14	1.403 (3)
S—C15	1.791 (2)	C10—C11	1.388 (3)
O1—C7	1.379 (2)	C10—H10	0.9500
O1—C8	1.383 (2)	C11—C12	1.383 (3)
C1—C8	1.361 (3)	C11—H11	0.9500
C1—C2	1.448 (3)	C12—C13	1.386 (3)
C2—C7	1.390 (3)	C12—H12	0.9500

C2—C3	1.400 (3)	C13—C14	1.388 (3)
C3—C4	1.382 (3)	C13—H13	0.9500
C3—H3	0.9500	C14—H14	0.9500
C4—C5	1.400 (3)	C15—H15A	0.9800
C5—C6	1.388 (3)	C15—H15B	0.9800
C5—H5	0.9500	C15—H15C	0.9800
C6—C7	1.379 (3)		
C4—I—O2 ⁱ	169.12 (6)	C1—C8—C9	135.0 (2)
O2—S—C1	107.16 (9)	O1—C8—C9	114.7 (2)
O2—S—C15	105.6 (1)	C10—C9—C14	119.5 (2)
C1—S—C15	98.5 (1)	C10—C9—C8	120.9 (2)
C7—O1—C8	106.8 (1)	C14—C9—C8	119.6 (2)
C8—C1—C2	107.3 (1)	C11—C10—C9	120.4 (2)
C8—C1—S	126.4 (2)	C11—C10—H10	119.8
C2—C1—S	126.0 (2)	C9—C10—H10	119.8
C7—C2—C3	119.1 (2)	C12—C11—C10	119.9 (2)
C7—C2—C1	105.2 (2)	C12—C11—H11	120.1
C3—C2—C1	135.7 (2)	C10—C11—H11	120.1
C4—C3—C2	117.2 (2)	C11—C12—C13	120.3 (2)
C4—C3—H3	121.4	C11—C12—H12	119.9
C2—C3—H3	121.4	C13—C12—H12	119.9
C3—C4—C5	122.6 (2)	C12—C13—C14	120.4 (2)
C3—C4—I	118.8 (1)	C12—C13—H13	119.8
C5—C4—I	118.6 (1)	C14—C13—H13	119.8
C6—C5—C4	120.6 (2)	C13—C14—C9	119.5 (2)
C6—C5—H5	119.7	C13—C14—H14	120.2
C4—C5—H5	119.7	C9—C14—H14	120.2
C7—C6—C5	116.1 (2)	S—C15—H15A	109.5
C7—C6—H6	122.0	S—C15—H15B	109.5
C5—C6—H6	122.0	H15A—C15—H15B	109.5
O1—C7—C6	125.1 (2)	S—C15—H15C	109.5
O1—C7—C2	110.4 (2)	H15A—C15—H15C	109.5
C6—C7—C2	124.5 (2)	H15B—C15—H15C	109.5
C1—C8—O1	110.3 (2)		
O2—S—C1—C8	-141.49 (17)	C1—C2—C7—O1	-0.9 (2)
C15—S—C1—C8	109.18 (19)	C3—C2—C7—C6	-0.5 (3)
O2—S—C1—C2	31.55 (19)	C1—C2—C7—C6	-179.18 (19)
C15—S—C1—C2	-77.78 (19)	C2—C1—C8—O1	0.0 (2)
C8—C1—C2—C7	0.5 (2)	S—C1—C8—O1	174.13 (14)
S—C1—C2—C7	-173.63 (14)	C2—C1—C8—C9	177.4 (2)
C8—C1—C2—C3	-177.8 (2)	S—C1—C8—C9	-8.5 (3)
S—C1—C2—C3	8.1 (3)	C7—O1—C8—C1	-0.6 (2)
C7—C2—C3—C4	-0.1 (3)	C7—O1—C8—C9	-178.51 (16)
C1—C2—C3—C4	178.1 (2)	C1—C8—C9—C10	-29.1 (3)
C2—C3—C4—C5	0.7 (3)	O1—C8—C9—C10	148.17 (18)
C2—C3—C4—I	-177.86 (14)	C1—C8—C9—C14	152.5 (2)
O2 ⁱ —I—C4—C3	40.4 (4)	O1—C8—C9—C14	-30.3 (3)
O2 ⁱ —I—C4—C5	-138.2 (3)	C14—C9—C10—C11	-0.6 (3)

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C3—C4—C5—C6	-0.7 (3)	C8—C9—C10—C11	-179.03 (19)
I—C4—C5—C6	177.79 (15)	C9—C10—C11—C12	0.4 (3)
C4—C5—C6—C7	0.2 (3)	C10—C11—C12—C13	0.0 (3)
C8—O1—C7—C6	179.20 (19)	C11—C12—C13—C14	-0.1 (3)
C8—O1—C7—C2	0.9 (2)	C12—C13—C14—C9	-0.1 (3)
C5—C6—C7—O1	-177.60 (18)	C10—C9—C14—C13	0.5 (3)
C5—C6—C7—C2	0.5 (3)	C8—C9—C14—C13	178.93 (18)
C3—C2—C7—O1	177.79 (16)		

Symmetry codes: (i) $-x+1, -y+1, -z$.

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C6-H6\cdots O1^{ii}$	0.95	2.59	3.536 (2)	173

Symmetry codes: (ii) $-x+1, -y, -z+1$.

Fig. 1

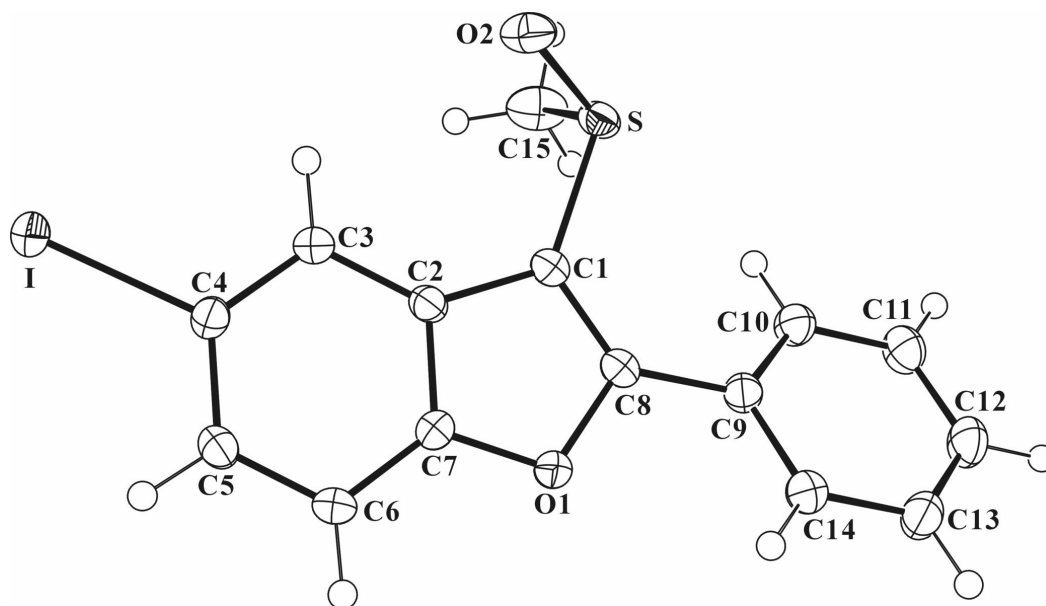


Fig. 2

